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# Mechanism of Ultrafine Grain Formation During Intense Plastic Straining in an Aluminum Alloy at Intermediate Temperatures

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## Abstract

The mechanism of grain formation during equal channel angular extrusion (ECAE) in a 2219 Al alloy has been studied at intermediate and high temperatures. It was shown that continuous dynamic recrystallization (CDRX) occurred during intense plastic straining and resulted in the formation of submicrometer grains at temperatures ranging from 250°C to 300°C. Higher temperatures (>300°C) hindered CDRX. This is caused by the fact that nucleation controls CDRX in the aluminum alloy. Dislocation rearrangements result in the formation of low angle boundary networks at moderate strain. The density of lattice dislocations determines the rate of subgrain formation. In addition, at lower temperatures a low energy dislocation structure (LEDS) forms concurrently with the subgrain structure and stabilizes it. The stability of the subgrain structure is very important for the resulting conversion of low angle boundaries into high angle ones with strain by extensive accumulation of mobile lattice dislocations. Increasing temperature in the range of intermediate temperatures suppresses LEDS formation and decreases the lattice dislocation density. This reduces the rate of the subgrain formation process and CDRX. As a result, at T=400°C no recrystallized grains were found. At T=475°C, the new grains form due to geometric dynamic recrystallization (GRX).

**Keywords:** 2219 aluminum alloy; Continuous dynamic recrystallization; Geometric dynamic recrystallization; Microstructure evolution; equal channel angular extrusion.

## 1. Introduction

It is known that ECAE results in the formation of an ultrafine grain structure in aluminum alloys [1-3]. However, only a limited number of studies have focussed on the evolution of microstructure during intense plastic deformation [1-6]. In a previous article by Kaibyshev et al. [3], it was shown that new grains formed during ECAE in pure aluminum and the 2219 alloy by a CDRX mechanism at room temperature and T=250°C, respectively. It was concluded in the Kaibyshev et al. study [3] (on the basis of inspection of results reported in references 2,5,6) that the formation of a LEDS after the first passage through the die during ECAE had an important influence on the formation of a submicrocrystalline structure in aluminum. This stabilization was a primary requirement for the onset of CDRX and formation of submicrometer scale grains in aluminum alloys. In the 2219 alloy, the secondary phase particles provide additional stabilization of the subgrain structure [3]. In addition, other factors affected the formation of an ultrafine grain structure during ECAE - especially temperature. The influence of these factors on grain formation is still unknown. This lack of knowledge hinders the understanding of grain formation mechanisms. The major objective of the present work is to study the influence of temperature on CDRX in the 2219 aluminum alloy and consider factors affecting grain refinement during intense plastic deformation.

## 2. Materials and Experimental Techniques

The material and experimental techniques used in the present study have been described elsewhere [3]. Samples of 2219 Al alloy were severely deformed by ECAE processing at several temperatures at a strain rate of  $10^{-2} \text{ s}^{-1}$ . The temperatures and true strains (reported in parenthesis) used during ECAE processing were 250°C ( $\epsilon=8$ ), 300°C ( $\epsilon=4$ ), 400°C ( $\epsilon=4$ ) and 475°C ( $\epsilon=12$ ).

## 3. Results

Microstructural evolution of the 2219 aluminum alloy during ECAE at  $T=250^\circ\text{C}$  has been previously reported in detail [3]. Increasing the temperature of processing up to  $300^\circ\text{C}$  results in changes in the character of microstructural evolution. The density of separate lattice dislocations,  $\rho=1.5 \cdot 10^9 \text{ cm}^{-2}$ , observed after the first passage through the die at  $T=300^\circ\text{C}$  is smaller than that at  $T=250^\circ\text{C}$  by a factor of 3.5. The first pass through the die results in the formation of a poorly-defined cell structure (Fig.1a). Low-angle boundaries are observed primarily in the vicinity of initial grain boundaries. With additional passes through the die, the dislocation density gradually increases with strain up to average values of  $4 \cdot 10^9 \text{ cm}^{-2}$  and  $8 \cdot 10^9 \text{ cm}^{-2}$  after  $\epsilon=2$  and  $\epsilon=4$ , respectively. After the second pass through the die, the formation of a mixed dislocation structure consisting of two structural components (Figs.1b and 1c) was observed. One component of the microstructure was a typical cell structure containing bands of equiaxed cells in which the boundaries have a high dislocation density and the interiors have a low dislocation density (Fig.1b). The other structural component was a well-defined subgrain structure (Fig. 1c) located between the cell bands. Dense dislocation walls (DDW) did not form at this temperature. Increasing the temperature of processing up to  $300^\circ\text{C}$  also results in the growth of (sub)grains formed during intense plastic deformation and a reduction in the volume fraction of LEDS formed after the first and second passes. Subgrains and cells are essentially equiaxed after a true strain  $\sim 2$ , and the average (sub)grain size is about  $1.1 \text{ }\mu\text{m}$  and the average cell size is  $0.7 \text{ }\mu\text{m}$ . The volume fraction of the subgrain structure is higher than that of the cell structure and exceeds 60–70%. After the fourth pass, a mixed network of high-angle and low-angle boundaries was observed (Fig.1d). Areas of elongated (sub)grains with an average size of  $2.1 \text{ }\mu\text{m}$  in the longitudinal direction and  $1.2 \text{ }\mu\text{m}$  in the transverse direction alternate with the equiaxed (sub)grain structure with an average size of  $1.6 \text{ }\mu\text{m}$ . This structure is indicative of the lower stability of the low-angle boundary network at  $T=300^\circ\text{C}$  relative to the network formed at  $T=250^\circ\text{C}$ , where the influence of strain on the average sub(grain) size was found to be insignificant. Notably after four passes through the die, the volume fraction of high-angles boundaries at  $T=300^\circ\text{C}$  is, at least, half that at  $T=250^\circ\text{C}$ .

Increasing the processing temperature to  $400^\circ\text{C}$ , suppresses the formation of the cell structure during intense plastic deformation. The average dislocation density slightly exceeds  $4 \cdot 10^9 \text{ cm}^{-2}$  at all strains. The first pass through the die results in the elongation of the initial grains. An equiaxed subgrain structure with average size of  $3.7 \text{ }\mu\text{m}$  forms after the second pass (Fig. 2a). Increasing strain results in a gradual increase in the deformation-induced boundary misorientation. However, even after a true strain  $\sim 4$  no true high-angle boundaries were observed (Fig.2b).

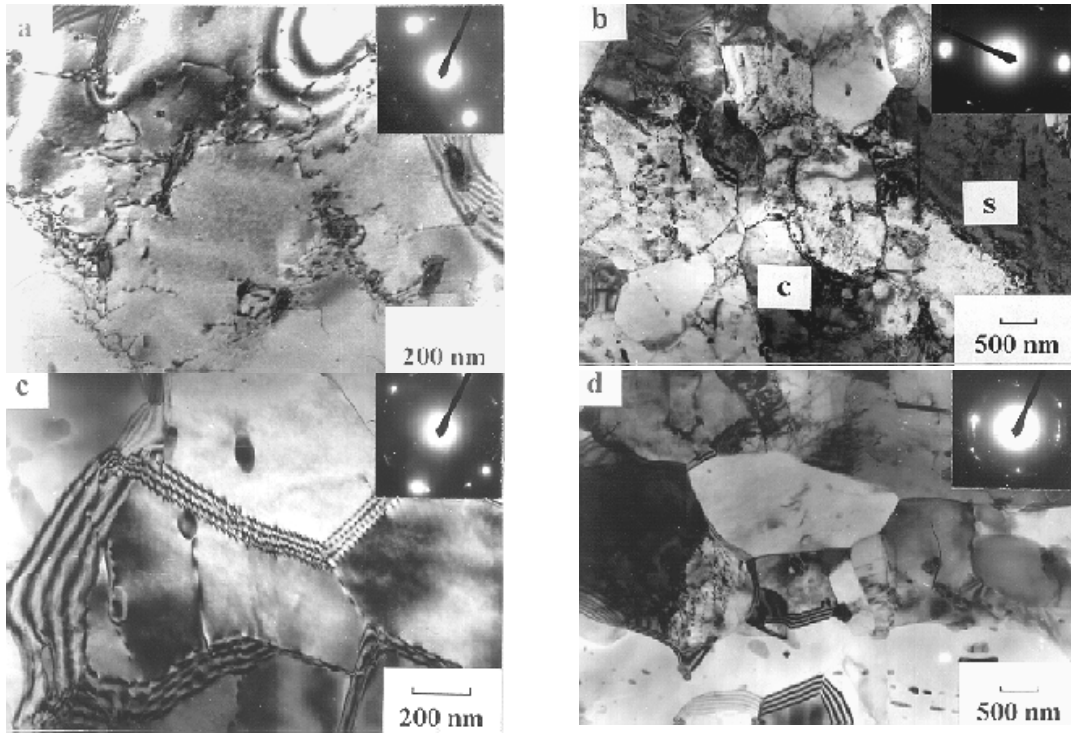


Fig. 1. Microstructures and corresponding SAED patterns of AA2219 after ECAE at  $T=300^{\circ}\text{C}$ : (a)  $\epsilon=1$ ; (b)  $\epsilon=2$ , s-subgrains, c-cells; (c)  $\epsilon=2$ ; (d)  $\epsilon=4$ .

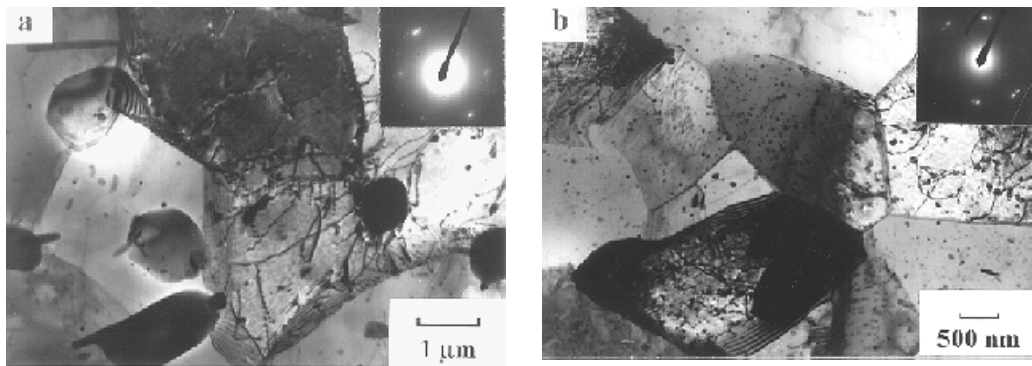
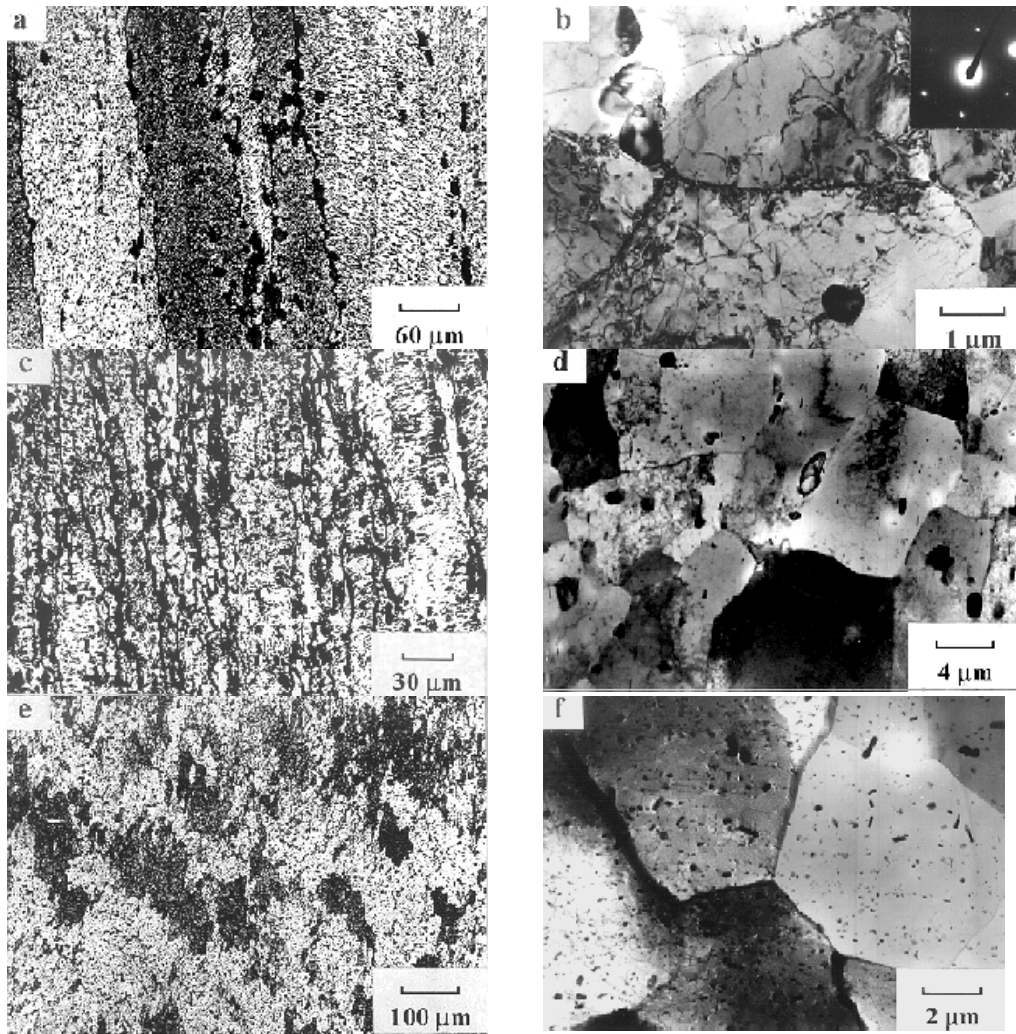


Fig.2. Microstructures and corresponding SAED patterns of AA2219 after ECAE at  $T=400^{\circ}\text{C}$ : (a)  $\epsilon=2$ ; (b)  $\epsilon=4$ .

At  $T=475^{\circ}\text{C}$ , the ECAE processing results in gradual elongation of the initial grains in the extrusion direction and serration of the initial boundaries (Fig.3a). Concurrently, the low-angle boundaries with misorientations less than  $1-2^{\circ}$  were formed inside the grains (Fig.3b). The density of separate lattice dislocations is about  $2 \cdot 10^9 \text{cm}^{-2}$ . Increasing deformation results in the gradual reduction in the transverse size of initial grains (Fig.3c). Low-angle boundaries were observed in the transverse direction and subdivide the volume of initial grains into separate crystallites. Subboundaries tie up the opposite grain boundaries (Fig.3d). Impingement of the serrated grains is observed after a true strain  $\sim 8$ . This process results in the formation of separate grains with essentially equiaxed shape and size of about  $10-40 \mu\text{m}$  (Fig. 3e,f). These crystallites exhibit similar orientations and belong to few orientation groups in whole material volume. Low-angled boundaries are observed inside the new grains. Such a structure is usually interpreted as being due to GRX [7].



*Fig.3. Microstructures of AA2219 after ECAE at  $T=475^{\circ}\text{C}$ : (a)  $\epsilon=1$ ; (b)  $\epsilon=2$ ; (c)-(d)  $\epsilon=8$ ; (e)-(f)  $\epsilon=12$ .*

The precipitates of secondary  $\theta$ -phase as well as dispersoids of transition metals were found to be very effective in stabilizing the substructure by pinning the subboundaries that formed and retarding high angle boundary migration at all deformation temperatures. As a result, a large number of particles were observed at (sub)grain boundaries (see Figs. 1 - 3).

#### 4. Discussion

Analysis of the present results shows that CDRX occurs in the 2219 aluminum alloy during intense plastic deformation in the temperature range  $250\text{-}400^{\circ}\text{C}$ . The process of grain formation during ECAE consists of three stages:

- (i) formation of a low angle boundary network after the first pass;
- (ii) conversion of low-angle boundaries into high-angle boundaries due to progressive accumulation of lattice dislocations;
- (iii) deformation-induced boundary migration resulting in an equilibrium boundary network configuration.

Temperature has a strong effect on CDRX. Increasing temperature hinders grain formation during intense plastic deformation of the 2219 alloy. At higher temperatures, the kinetics of CDRX are also slower, which results primarily from two factors. First, increasing

temperature results in uniform deformation and reduced tendency to form a LEDS. The LEDS plays an important role in the stabilization of the subgrain structure at  $T=250^{\circ}\text{C}$  [3]. At a temperature of  $300^{\circ}\text{C}$  the stabilization effect of the LEDS is reduced. This is caused by a decrease in the volume fraction of the LEDS and formation of only a cell structure. As a result, the formation of recrystallized grains at this temperature shifts toward higher strains. At  $T=400^{\circ}\text{C}$  no elements of a LEDS form, and the stabilization of the subgrain structure is provided by secondary particles, only. The instability of low-angle boundaries inhibits their transformation into high-angle boundaries due to trapping of mobile lattice dislocation. Therefore, the suppression of the LEDS formation with increasing temperature results in reduced stability of the low angle boundary network and reduced occurrence of CDRX. At  $T=475^{\circ}\text{C}$ , the progressive dissolution of  $\theta$ -phase substantially reduces the stability of the subgrain structure formed. As a result, no CDRX occurs because the subgrain structure is unstable inside the initial grains. At this higher temperature new grain formation occurs by GRX.

The second source of slower CRDX kinetics at higher temperatures is the reduction of the equilibrium dislocation density, which hinders the formation of low angle boundaries by dislocation rearrangement. It was shown by Kaibyshev et al. [8] that warm deformation occurs in the temperature range  $250\text{-}400^{\circ}\text{C}$ . Increasing the temperature in this interval significantly enhances dislocation climb. As a result, mutual annihilation of lattice dislocations due to dynamic recovery is accelerated and the dislocation density decreases. This decreases the probability for low angle boundary formation.

The relationship between dislocation density, subgrain size and subboundary misorientation can be calculated for cubic-shaped subgrains that result from lattice dislocation rearrangements of dislocation pile-ups. The dislocation density,  $\rho_0$ , is equal to the total dislocation length,  $L$ , in a volume unit,

$$\rho_0 = L/d^3, \quad (1)$$

where  $d$  is the length of the cube face (subgrain size). We obtain for a cube

$$L = 6 \cdot d \cdot n, \quad (2)$$

where  $n$  is number of dislocations in the boundary. Thus,

$$\rho_0 = 6 \cdot d \cdot n/d^3 = 6 \cdot r/d, \quad (3)$$

where  $r = n/d$  is the linear dislocation density in a subboundary. It is known [9], that

$$r = (\Theta/b) \cdot (\pi/180^{\circ}), \quad (4)$$

where  $b$  is the Burgers vector of a dislocation and  $\Theta$  is the misorientation of the subboundary (taken in degrees). Combining Eqns. (3) and (4) yields the following expression for the relationship between the lattice dislocation density and the misorientation of a deformation-induced boundary,

$$\rho_0 = 6 \cdot [\Theta/(d \cdot b)] \cdot (\pi/180^{\circ}). \quad (5)$$

Equation (5) has been evaluated for reasonable values of misorientation ( $\Theta = 0.1^{\circ} - 15^{\circ}$ ), subgrain size ( $d = 0.1 \mu\text{m} - 15 \mu\text{m}$ ) and Burgers vector ( $b = 2.8 \cdot 10^{-10} \text{ m}$  for Al [8]). Fig. 4 shows the relationship between dislocation density, crystallite size and misorientation of

boundaries surrounding a crystallite. It is seen that a reduction in the lattice dislocation density results in the growth of the crystallite or a decrease in boundary misorientation. However, misorientation of a stable deformation-induced boundary can not be less than  $1-2^\circ$ . A boundary with lower misorientation is unstable and can not increase its misorientation due to the trapping of mobile dislocations. Thus, dynamic recovery, which results in a decrease in lattice dislocation density, hinders CRDX.

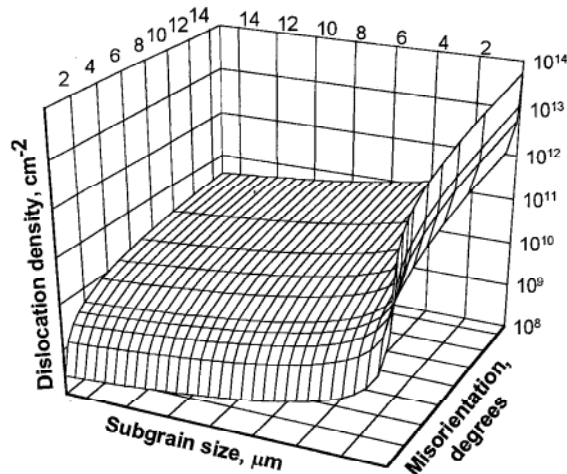


Fig.4. Average dislocation density vs subgrain size and subgrain boundary

In summary, ultrafine grains form during intense plastic deformation in the 2219 aluminum alloy over a range of intermediate temperatures due to CDRX. Dislocation rearrangements are an important component of the CDRX mechanism. On one hand dislocation rearrangements result in the formation of a LEDS, which provides stability to the evolving subgrain structure. The enhanced subboundary stability resulting from the LEDS is a requirement for a high rate of misorientation growth and rapid kinetics of conversion of low-angle boundaries into true high-angle boundaries. On the other hand, the higher lattice dislocation density accelerates the formation of a low-angle boundary network after the first pass

through the die. Such a structure transforms into a recrystallized granular structure at a very high rate. Increasing temperature provides increased dislocation mobility by climb, which suppresses the formation of the LEDS and hinders dislocation accumulation inside the initial grains due to mutual annihilation. As a result, CDRX is impeded by increasing temperature and does not occur at  $T=475^\circ\text{C}$ . The formation of coarser crystallites at higher temperatures is associated with a reduction in the accumulated dislocation density. Submicrometer-scale grains may form in the 2219 aluminum alloy during intense plastic deformation in the transition interval between warm and cold deformation.

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